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MINIMIZING POLLUTION IN CLEANING AND DEGREASING OPERATIONS

A.R. Tarrer, H.J. Sanjay - Auburn University
S. Garry Howell - U.S. EPA

ENVIRONICS DIRECTORATE
139 Barnes Drive, Suite 2
Tyndall AFB FL 32403-5323

Auburn University
Department of Chemical Engineering
230 Ross Hall
Auburn University AL 36849-5127

US EPA/RREL
26 W Martin Luther King Drive
Cincinnati OH 45268

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TYNDALL AIR FORCE BASE, FLORIDA 32403-5323

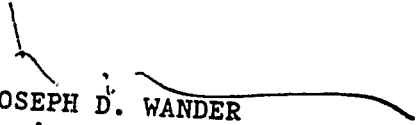
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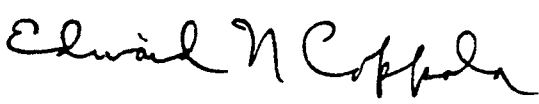
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JOSEPH D. WANDER
Project Officer


NEIL J. LAMB, Col, USAF, BSC
Chief, Environics Division


EDWARD N. COPPOLA, Major, USAF
Chief, Environmental Compliance
Division

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13. (continued) while the acid acceptance value (AAV) remained essentially constant. Slight deviations from best operating techniques and minor obstructions in return lines or overheating of evaporation coils caused significant increases in evaporative losses. The study shows that recovery by purification and distillation and in-situ filtration are practical options to extend the effective lifetime of metal-cleaning and degreasing solvents when used in conjunction with a program to monitor and maintain levels of protective additives. Significant refinement of the in-situ filtration process will be needed before implementation can be recommended.

PREFACE

This report was prepared by Auburn University, Department of Chemical Engineering, 220 Ross Hall, Auburn University AL 36849-5127, under contract #EPA-CR-816219-01, for the U.S. Air Force, Armstrong Laboratory Environics Directorate (AL/EQS-OL), 139 Barnes Drive, Suite 2, Tyndall AFB FL 32403-5323, and the U.S. Environmental Protection Agency Risk Reduction Engineering Laboratory (EPA/RREL), 26 W Martin Luther King Drive, Cincinnati OH 45268.

This technical report summarizes work done between 14 February 1989 and 10 October 1991. The EPA project officer was Mary Ann Curran of RREL; the Air Force project officers were Surendra B. Joshi of HQ AFESC/RDVS and Dr. Joseph D. Wander of AL/EQS.

Generous cooperation by Air Force personnel at Warner Robins Air Logistics Center (WR-ALC) and Tinker Air Force Base (OC-ALC) is gratefully acknowledged.

EXECUTIVE SUMMARY

A. OBJECTIVE

The objective of this study was to examine approaches to decreasing rates of loss by evaporation and to extend the useful lifetime of metal-cleaning solvents in service as means to decrease the generation of pollutant emissions and residues from Air Force cleaning and degreasing operations.

B. BACKGROUND

In an earlier study, several properties of cleaning solvents were measured at intervals during usage and correlated with the cleaning ability of the solvent. The purpose of that exercise was to extend the lifetime of the solvent by developing quantitative measures of the extent of contamination or of expenditure of metal-protecting additives. Application of the endpoints identified would actually remove the solvents from service before their cleaning capability had been exhausted. Of these tests, the acid acceptance value (AAV)--which measures the amount of acid inhibitor remaining--was the most reliable. Field experience has shown that replacement of the inhibitors can restore usability to partially contaminated 1,1,1-trichloroethane (TCA).

C. SCOPE

This report includes data from an experimental study in which the same properties identified in the previous study were measured on solvents that had been removed from service for recovery. The used solvents were pumped from a sump through a mechanical filter for a period of time corresponding to five complete passages through the filter, and the evolution of the properties was followed as a function of the extent of treatment. A second section contains an evaluation of the operation and maintenance of several degreasers in operation at Tinker AFB, together with specific suggestions to decrease the rate of evaporative loss from these units.

D. METHODOLOGY

No new methods were used in this study. Water content was measured by a Karl Fischer method (ASTM 1364-90). AAV was measured by addition of an aliquot of acid and back-titration with alkali (ASTM D-2942). Visible transmittance was measured with a direct-reading UV--visible spectrometer, electrical conductivity with a conductivity meter, and viscosity with a falling-ball viscometer. Nonvolatile matter (NVM) was measured gravimetrically, either by evaporating the solvent over a steam bath or by filtering and evaporating the adhering solvent. The survey of the degreasers was performed by Jim C. Johnston of Dow Chemical Company, U.S.A.

E. TEST DESCRIPTION

A measured volume of spent TCA was delivered into the sump of the recirculating filtration system. Small samples were withdrawn for analysis and the solvent was circulated through a pair of filters in tandem and back into the sump. Sampling was repeated at intervals corresponding to the time required to pump the entire volume of solvent through the filters for five cycles. The evaluation of the degreasers was a walk-through inspection that included no quantitative measurements.

F. RESULTS

During recirculation through the filter, water content and color intensity (from transmittance measurements) decreased with reasonable consistency, while AAV remained essentially constant. There is some inconsistency and scatter in the data, but the trends are consistent. (At the end of the treatment, the inhibitors are preserved and the general condition of the solvent is improved enough for preliminary cleaning.) Slight deviations from best operating techniques and minor obstructions in return lines or overheating of evaporation coils cause significant increases in the rate of evaporative loss from average-to-good degreasing operations.

G. CONCLUSIONS

Recovery by purification and distillation and in-situ filtration are practical options to extend the effective working lifetime of metal-cleaning and degreasing solvents when used in conjunction with a program to monitor levels of protective additives and maintain additive protection above critical thresholds. Significant refinement of the in-situ filtration process will be needed before implementation can be recommended. Refinement of operating technique and aggressive maintenance policies can decrease the rate of loss of solvent from degreasing baths.

H. RECOMMENDATIONS

The following recommendations are offered: (1) Refine and expand in-situ purification technology and implement as a field test, (2) Retrain degreaser operators at regular intervals, and (3) Conduct unscheduled evaluations of operating technique on the line and of maintenance status of the degreasing baths.

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SECTION I

INTRODUCTION

A. PROJECT OBJECTIVES

Large volumes of chlorinated solvents are used in cleaning and degreasing U.S. Air Force aircraft and engine parts. The objective of this project is to investigate methods to minimize loss of solvents through evaporation and extend the useful lifetime of metal-cleaning solvents in use at overhaul and maintenance operations.

B. BACKGROUND

Overhaul and maintenance operations operated by the U.S. Air Force use large volumes of solvents for cleaning and degreasing of aircraft and engine parts, gyroscopes, guidance systems, and electronic components. A major portion of these solvents are chlorinated compounds such as 1,1,1-trichloroethane, methylene chloride, or perchloroethylene. During normal use, some of these materials evaporate into the atmosphere, and are thought to contribute to stratospheric ozone depletion. As grease and dirt build up in cleaning baths, a point is reached at which the solvent must be discarded, resulting in the loss of the value of the solvent, plus the added cost of disposal. As part of the implementation of its waste minimization program, the U.S. Air Force is trying to find the most efficient, safe, and economical methods to extend the useful life of (or recycle) cleaning solvents, particularly TCA. Other less widely used solvents considered for recycling are methylene chloride, perchloroethylene, and PD-680 (a petroleum fraction similar to the well known Stoddard solvent). Properties of some common cleaning solvents are given in Table 1.

C. SCOPE

Auburn University, in cooperation with major solvent manufacturers, developed a research program to determine which physical and chemical properties were most important for maximizing the useful life of the solvents. The target was to develop fairly simple methods for determining these properties so that users could get the longest useful life from their cleaning baths. After testing has shown that the the solvent is no longer usable, two options are offered: (1) critical additives may be replenished, or (2) the solvent may be recycled by distillation, filtration, or other means.

The first sections of this report concentrate on the TCA recycling technology developed at Auburn University by Professor A.R. Tarrer (Reference 1). Subsequent sections will address other solvents and their potential for recycling.

Efficient operation of degreaser baths can also reduce solvent usage and minimize emissions. An expert from Dow Chemical Co. was asked to audit degreasing operations at Tinker AFB, Oklahoma, and recommend changes that would accomplish these goals.

TABLE 1. COMMONLY USED DEGREASING SOLVENTS

<u>Hydrocarbons^b</u>	<u>Boiling Range</u>		<u>Flash Point^a</u>		<u>Uses^c</u>
	<u>°C</u>	<u>°F</u>	<u>°C</u>	<u>°F</u>	
Mineral Spirits	116-135	240-275	10	50	D, S
Aromatic Naphtha	200-350	392-660	65	150	D, S
Stoddard Solvent	155-210	310-410	<38	<100	D, S
Toluene	111	231	4	40	D, S
Xylene	135-143	275-289	85	185	D, S
<u>Alcohol</u>					
2-Propanol (Isopropyl Alcohol)	82	180	21	70	D, S
<u>Ketones</u>					
Acetone	55-57	131-135	-20	-4	D, S
Methyl Ethyl Ketone (MEK)	80	176	-4	25	D, S
<u>Halogenated Solvents</u>					
Methylene Chloride	40	104	NF ^d		D, S, V
Perchloroethylene (perc)	121	250	NF		D, S, V
1,1,1-Trichloroethane (TCA) ^e	74	165	NF		D, S, V
Trichloroethylene (TCE)	87	188	NF		D, S, V
Trichlorotrifluoroethane (CFC-113) ^f	48	118	NF		D, S, V

^a Typical range

^b With the exception of toluene and xylene, these solvents are petroleum fractions having a range of molecular weights, and consequently a boiling range, rather than discrete boiling points. Xylene, when used as a solvent, is usually a mixture of *ortho*, *meta*, and *para* isomers, each having a discrete boiling point.

^c D = dip, S = spray or brush, V = vapor

^d NF = Non Flammable

^e Also called methyl chloroform

^f Also available as binary azeotropes with ethanol, 2-propanol, acetone, and methylene chloride. Several ternary azeotropes are also available.

SECTION II

DEGREASING SOLVENTS

A. PHYSICAL CHANGES

Vapor degreasing is a common industrial process in which the parts to be cleaned are suspended in the vapor space above a boiling bath of solvent. The most common solvents for such operations are TCA and perc. The vapors are much heavier than air, so that a properly designed and operated bath (see Reference 2) will confine most of the vapors in the "head space" and lose very little to the atmosphere. During operation, solvents in a vapor degreaser undergo both chemical and physical changes. In addition, the liquid in the boiling sump starts to build up solids such as metal chips, dirt, or paint particles. Oils and greases are in solution in the liquid phase, and may build up sufficiently to increase viscosity and seriously impair heat transfer; in electrically heated baths, heater sheath temperatures may increase and cause failure. As the concentration of lighter oils increases, they will appear in the vapors, and will leave a noticeable residue on the "cleaned" parts. A chemical analysis aimed only at the composition of the solvent itself may show that it is still usable, but its efficiency is severely impaired by dissolved material. At least part of this efficiency may be restored by some physical methods described in a later section.

B. CHEMICAL CHANGES

Partially chlorinated solvents tend to decompose after prolonged heating. This is manifested by chlorine breaking off the hydrocarbon backbone, forming hydrogen chloride (HCl). The HCl then combines with any water present to form hydrochloric acid, which causes corrosion of metals. (Fluorine, as in CFC-113, is more tightly bound, and less likely to be released). The HCl itself promotes further dechlorination; this is inhibited by additives called *acid acceptors*, which are designed to combine with or neutralize HCl. Table 2 lists some commonly used inhibitors (see Reference 3 for a review of inhibitor patents).

Many halogenated solvents are relatively resistant to attack by oxygen until a chlorine is abstracted. Removal of the chlorine (or fluorine) and an adjacent hydrogen leaves a double bond, which is susceptible to oxidation, particularly in the presence of metals such as copper or vanadium. Perchloroethylene and trichloroethylene have these double bonds already in place, and are more susceptible to oxidation. Another class of stabilizers called *oxidation inhibitors* or *antioxidants* may be added to retard this mode of decomposition.

The interaction between acid and metal can cause etching or corrosion of a metal surface. Conversely, as stated above, some metals promote oxidation, requiring some means of protecting the solvent from the metal. *Metal stabilizers* accomplish this by forming a protective film on metal surfaces that retards attack by acids, while deactivating the catalytic properties of the surface.

The concentration of these stabilizers in halogenated solvents is critical, particularly the ratio of metal stabilizer to acid acceptor. While other stabilizers decompose chemically, or react with acids and become depleted, metal stabilizers are intentionally chosen to be somewhat volatile to maintain some presence in the vapor phase. An almost monomolecular layer is formed on the metal surfaces so that, as the parts are withdrawn, adherent stabilizer is consumed.

TABLE 2
TYPICAL INHIBITORS/STABILIZERS AND THEIR FUNCTIONS

Function	Compound and Formula
Acid Acceptor ^{a,b}	Butylene Oxide (1,2-Epoxybutane) $\begin{array}{c} \text{CH}_3\text{CH}_2\text{CHCH}_2 \\ \diagup \quad \diagdown \\ \text{O} \end{array}$
Acid Acceptor ^a	Epichlorohydrin (1,2-Epoxy-3-chloropropane) $\begin{array}{c} \text{ClCH}_2\text{CHCH}_2 \\ \diagup \quad \diagdown \\ \text{O} \end{array}$
Acid Acceptor ^a	Butoxymethyloxirane (1,2-Epoxy-3-butoxypropane) $\begin{array}{c} \text{C}_4\text{H}_9\text{OC}_2\text{CH}_2\text{CHCH}_2 \\ \diagup \quad \diagdown \\ \text{O} \end{array}$
Metal Stabilizer ^{a,c}	1,4-Dioxane (1,4-Dioxacyclohexane) $\begin{array}{c} \text{CH}_2\text{CH}_2 \\ \diagup \quad \diagdown \\ \text{O} \quad \text{O} \\ \diagdown \quad \diagup \\ \text{CH}_2\text{CH}_2 \end{array}$
Antioxidant	Ionol ^{TM,d} , BHT (2,6-Di- <i>t</i> -butyl- <i>p</i> -cresol) $2,6-(\text{C}_4\text{H}_9)_2-4-\text{CH}_3\text{C}_6\text{H}_3\text{OH}$
Antioxidant ^b	Nitromethane CH_3NO_2

^a Flammable and classed as possible carcinogens.

^b Donahue et al. (Reference 1) misinterpreted mass spectral data and mistakenly identified nitromethane and α -butylene oxide (1,2-epoxybutane) as *N*-methoxymethanamine ($\text{CH}_3\text{ONHCH}_3$) and formaldehyde dimethylhydrazone ($\text{H}_2\text{C}=\text{NN}(\text{CH}_3)_2$), respectively, during analytical characterization of the additives present in TCA.

^c Fatty acid amines are sometimes used; these give some rust inhibition after parts are exposed to air.

^d Trademark Shell Oil Company. Ionol or BHT is illustrative of a broad class of phenolic antioxidants; it may not be suitable for all types of degreasing baths.

SECTION III

STABILIZER ANALYSIS

Visual inspection alone is unreliable as a means of determining the condition of degreasing solvents. A bath which is perfectly clear could be deficient in inhibitor, and one which is badly discolored can still be well stabilized. The most reliable method of determining the levels and types of stabilizers in halogenated solvents is by gas chromatography/mass spectrometry (GC/MS); however, the expense involved makes it impractical for most users. Dr. Tarrer has suggested several reliable methods of analyzing for stabilizers and for estimating the remaining useful life of a degreasing bath; when in doubt, one should use two or more of them. The acid acceptance value (AAV) is the most important parameter, as it is an accepted measure of stabilizer content (References 4 and 5). Viscosity is affected by dirt and grease buildup, and might be the second most important analysis, although conductivity is considered by some to be at least as important. Some operators of degreasers concur with Dr. Tarrer in principle but consider his criteria for judging bath condition overly conservative. They state that using his measurement methods, but lowering the standards, allows them to extend useful life of the solvent while still getting efficient cleaning.

A. ANALYSIS METHODS

1. Water Content

The presence of water is detrimental in two respects: first, it promotes corrosion at metal interfaces; second, it promotes hydrolysis of the halogen--carbon bond, releasing free HCl, which immediately combines with water to form hydrochloric acid and become even more corrosive. The accepted standard test for water content of volatile solvents is ASTM 1364-90, a Karl Fischer-type analysis.

2. Acid Acceptance Value (AAV)


The AAV is a measure of the concentration of acid acceptor in the solvent. It is easily determined by a simple titration as outlined in ASTM D-2942. Figure 1 is a calibration chart for AAVs with Dr. Tarrer's recommended minimum (Reference 2) for 1,1,1-trichloroethane before it must be redistilled or discarded.


3. Visible Absorbance

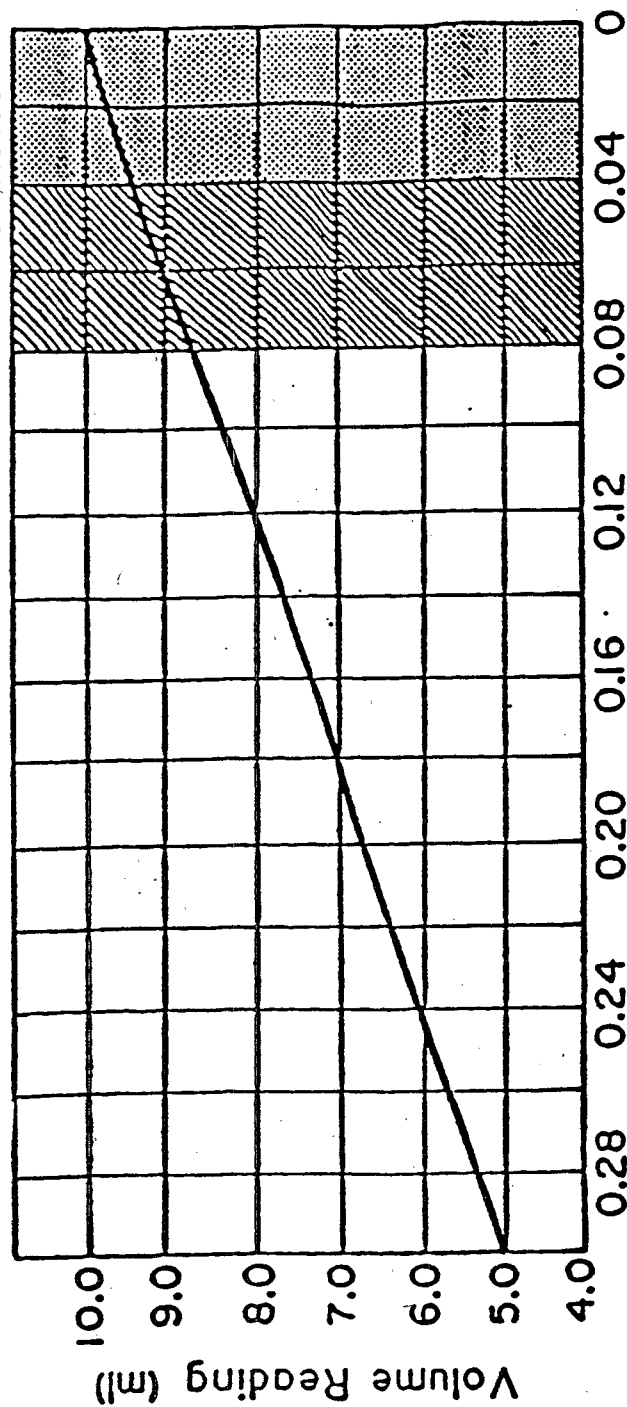
A spectrophotometer for absorbance testing can be bought for about \$2000; these instruments do not require extensive operator training.

1,1,1-TRICHLOROETHANE

Solvent Condition

 Borderline
-Check Daily

 Solvent Condition
Unacceptable
-Discard or Distill
for Reuse



Acid Acceptance as Weight Percent Sodium Hydroxide

Figure 1. 1,1,1-Trichloroethane Calibration Chart

4. Nonvolatile Matter (NVM)

While the accepted method is ASTM D-2109-85, evaporating solvent from a weighed sample over a steam bath will give good enough results for most users. The residue is oil, grease, soils, etc., which build up and impair cleaning efficiency.

5. Electrical Conductivity

Halogenated solvents are good insulators, and water containing ionic materials such as acids or salts will raise the conductivity measurably. Conductivity meters cost from \$750 to \$1500, and are as easily operated as spectrophotometers.

6. Viscosity

Many types of viscometers are available; a simple falling ball type can be bought from laboratory supply houses for about \$150.

SECTION IV

BATH REJUVENATION BY FILTRATION

As a degreasing or cleaning bath is used, and dissolved oil and grease, dirt, metal particles, and water accumulate, some steps must be taken to return it to a usable condition. Some of the tests run above might indicate that there is some useful life left, and that less drastic methods than discarding or distillation might suffice to extend it. An investigation of alternative rejuvenation methods was made by Professor Tarrer at Auburn University¹. He demonstrated that one approach is to carefully filter the solvent and monitor changes in properties by several of the methods noted above. An experimental filtration setup is illustrated in Figure 2. The solvent was pumped from a stirred tank through a series of three filters, the first being a bag that removed very coarse (25-micrometer) particles, then through 1.0- and 0.5-micrometer filters. Pumping was continued until the tank's contents were passed through the filters approximately five times; a single cycle was about 10 minutes. Samples were taken after each cycle to monitor the condition of the solvent. Filters were cleaned when the differential pressure across them reached 15 psi. Some test results from these filtration experiments are given below.

A. FILTRATION OF 1,1,1-TRICHLOROETHANE

1. Water Content

As stated before, the presence of water is deleterious to the solvent and to parts being cleaned. If the starting content was above about 0.5 percent, repeated filtration cycles actually reduced it as illustrated in Figures 3 and 4. In Runs 1, 2, and 5 in Figure 3, in which the initial concentrations were less than 0.5 percent, water content remained almost unchanged despite the number of passes through the filtration system. The reason for this is unclear, but may possibly be due to the fact that water solubility in TCA is about 500-600 ppm (0.05-0.06 percent), and above this level, as in Runs 9 and 10, and all the runs in Figure 4, the water exists as a dispersion. At higher water contents, filtration would cause some coalescence of droplets, causing them to float to the surface of the feed tank and evaporate. Whatever the mechanism, filtration done in this manner does remove some of the water.

¹ Performed as part of an Interagency Agreement between E.P.A. and the U.S. Air Force, which provided most of the funding. Dr. Joseph D. Wander of AFCESA/RAVS (now AL/EQS) was the Air Force project officer, and Mary Ann Curran of the Risk Reduction Engineering Laboratory was the EPA project officer on contract no. EPA-CR-816219-01.

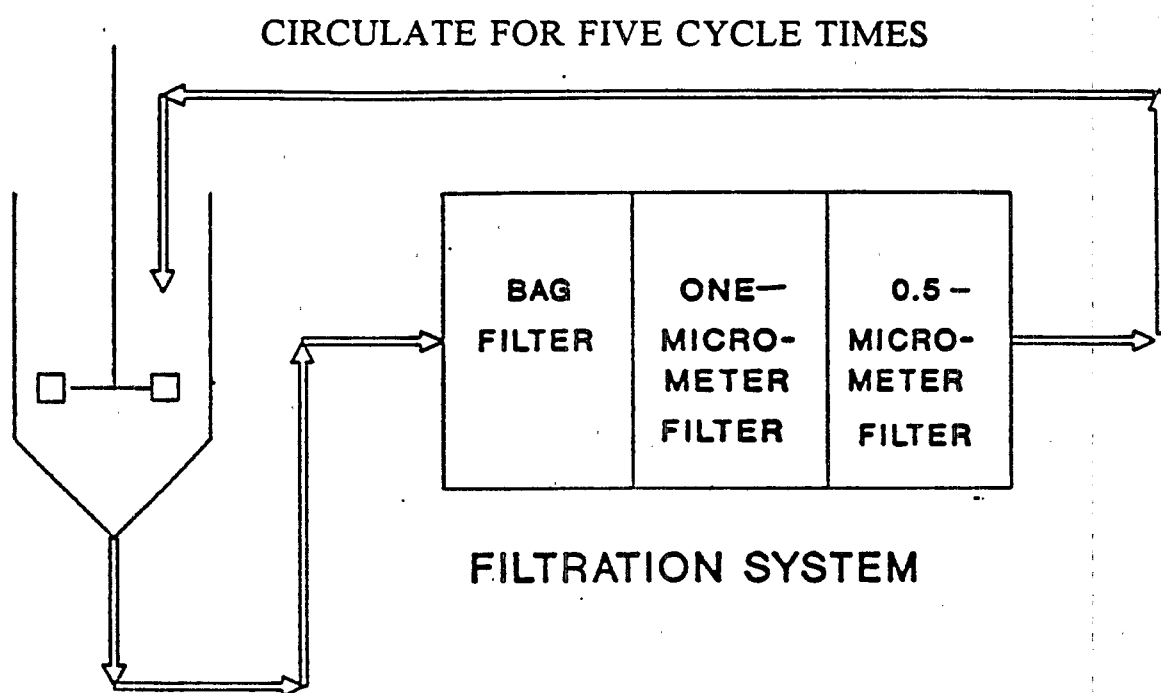


Figure 2. Filtration Scheme for Reclaiming Solvents

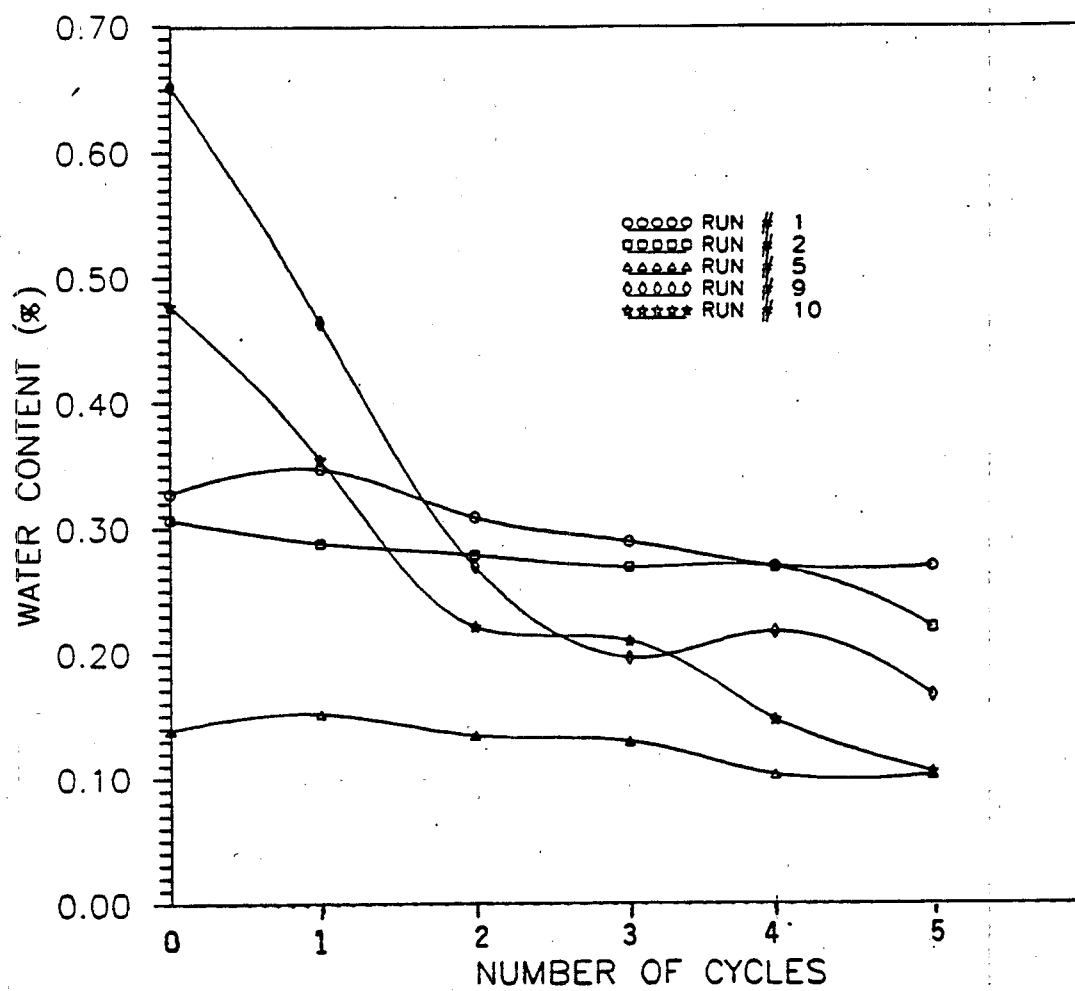


Figure 3. Water Content of 1,1,1-Trichloroethane for Various Cycle Times (Set #1)

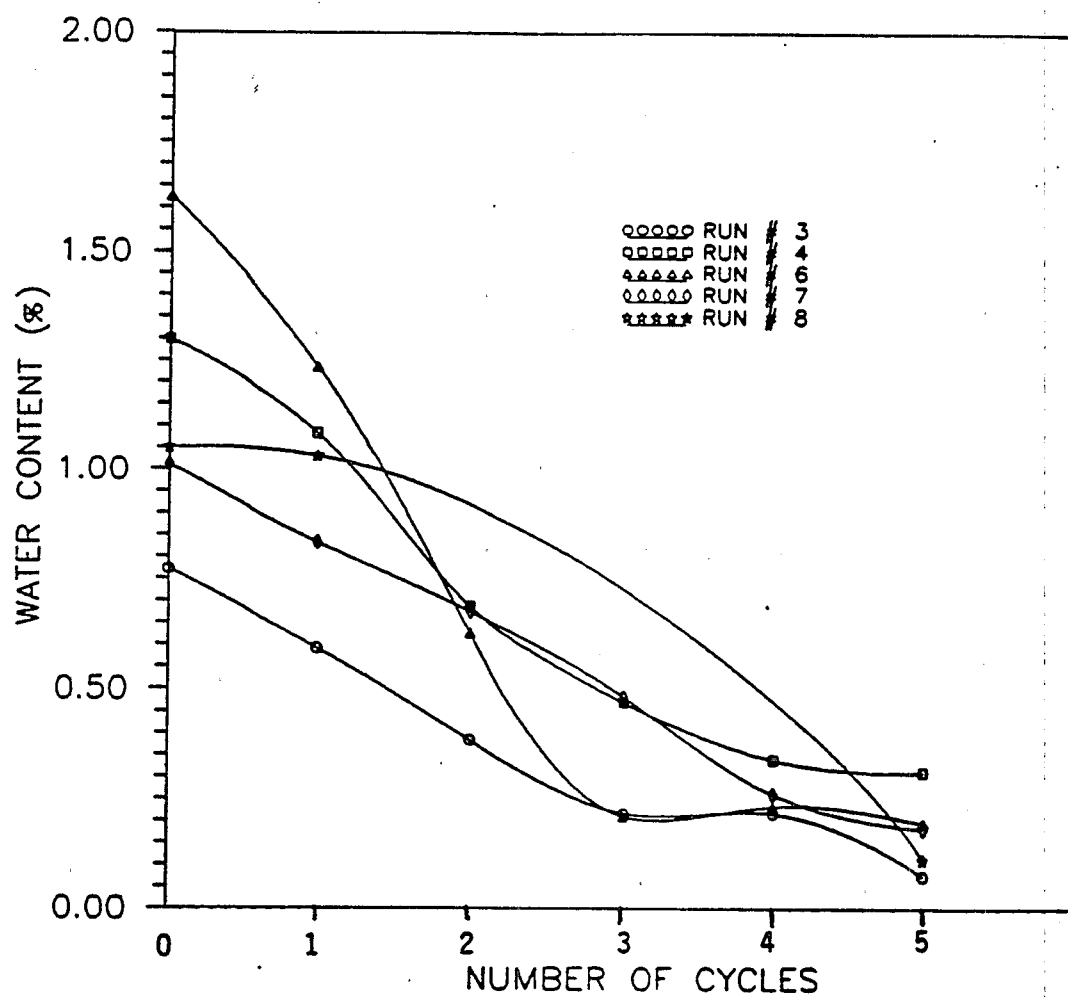


Figure 4. Water Content of 1,1,1-Trichloroethane for Various Cycle Times (Set #2)

2. Acid Acceptance Value (AAV)

The presence of acid acceptors (as measured by AAV) was not affected by filtration, as shown in Figures 5 and 6. Five passes through the system left the AAV almost unchanged at three times the minimum value. This is in contrast to distillation or adsorption on carbon, both of which decrease AAV.

3. Visible Absorbance

New TCA is water-white and crystal-clear, and its clarity is decreased by either dissolved materials or suspended particles. A portable spectrophotometer was used to measure the light (580 nm) absorbed or scattered by the solvent samples; this is reported as percent transmittance in Figures 7 and 8. Although water droplets would always be expected to have some effect on the light transmitted (or absorbed), the presence of water-soluble or wettable impurities can affect absorbance; however, the lack of correlation between Figs. 7 and 8 and Figs. 3 and 4, suggests that other factors could combine to increase absorbance. Dissolved colored waxes, oils, and grease as well as suspended particles of dirt, etc., also lower light transmission. Absorbance measurements should always be used in tandem with at least one other test, because a clear, clean-looking solvent could be completely devoid of acid acceptors or other stabilizers.

4. Nonvolatile Matter (NVM)

Weighed samples of contaminated solvent were evaporated under vacuum (unspecified) at $105 \pm 5^\circ\text{C}$. The NVM remained almost constant during all the runs. Measuring the solids content by filtering the samples, then drying the filter paper at 60°C gave higher values than the NVM. This was attributed to volatilization of lighter oils in the vacuum oven. Evaporating the samples on a steam table (the preferred method) would probably give higher results than the filter paper method, particularly if dissolved solids were present.

5. Conductivity

Electrical conductivity tests made after every pass through the filtration system showed very little variation; however, comparison to virgin solvent indicated contamination by some conductive material. The small amount of water dissolved in the TCA (the literature values are 500-600 ppm) could carry minute amounts of HCl or salts, which would raise the conductivity well above the 10^{-10} mhos/cm of the new material. Filtration, as stated previously, will not remove dissolved materials, so little or no effect on conductivity would be expected.

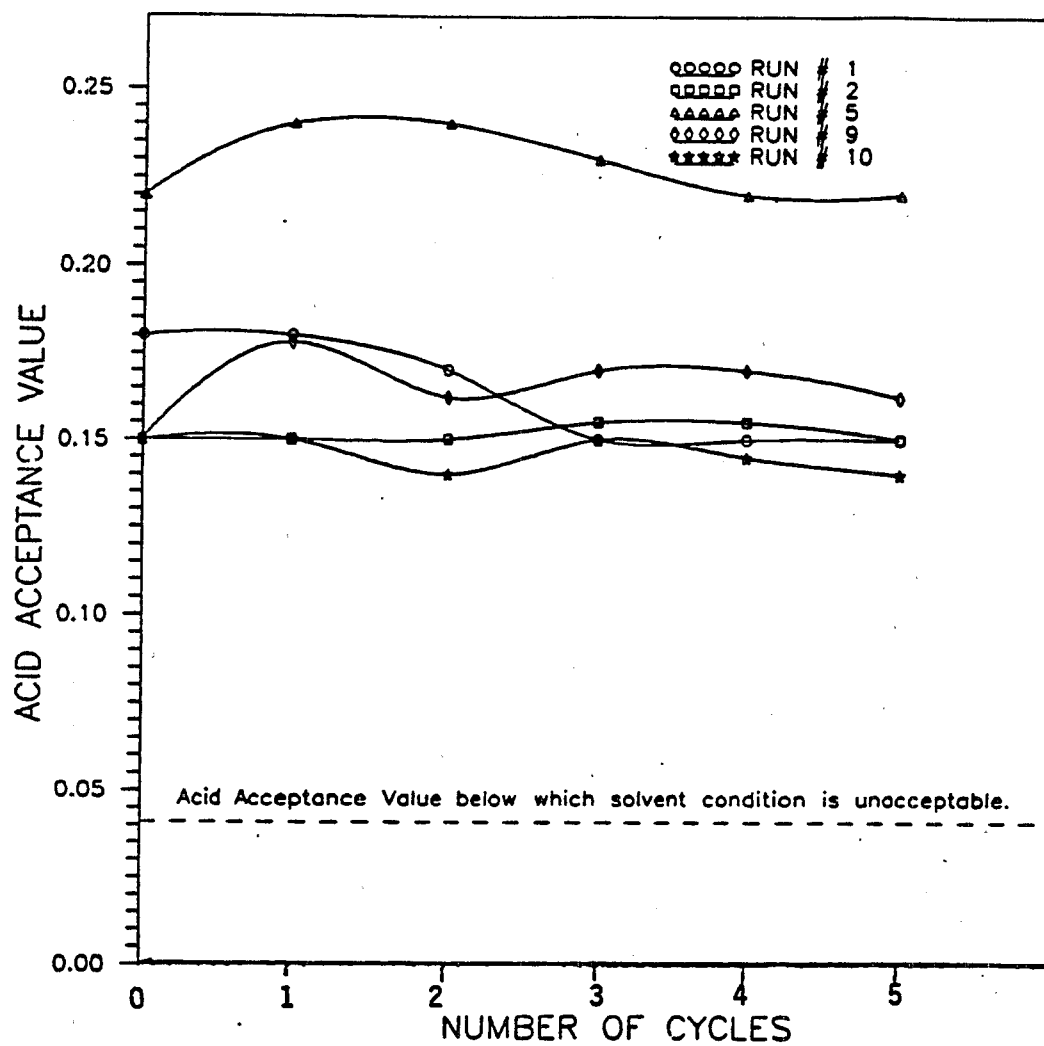


Figure 5. Variation of Acid Acceptance Value With Cycle Times (Set #1)

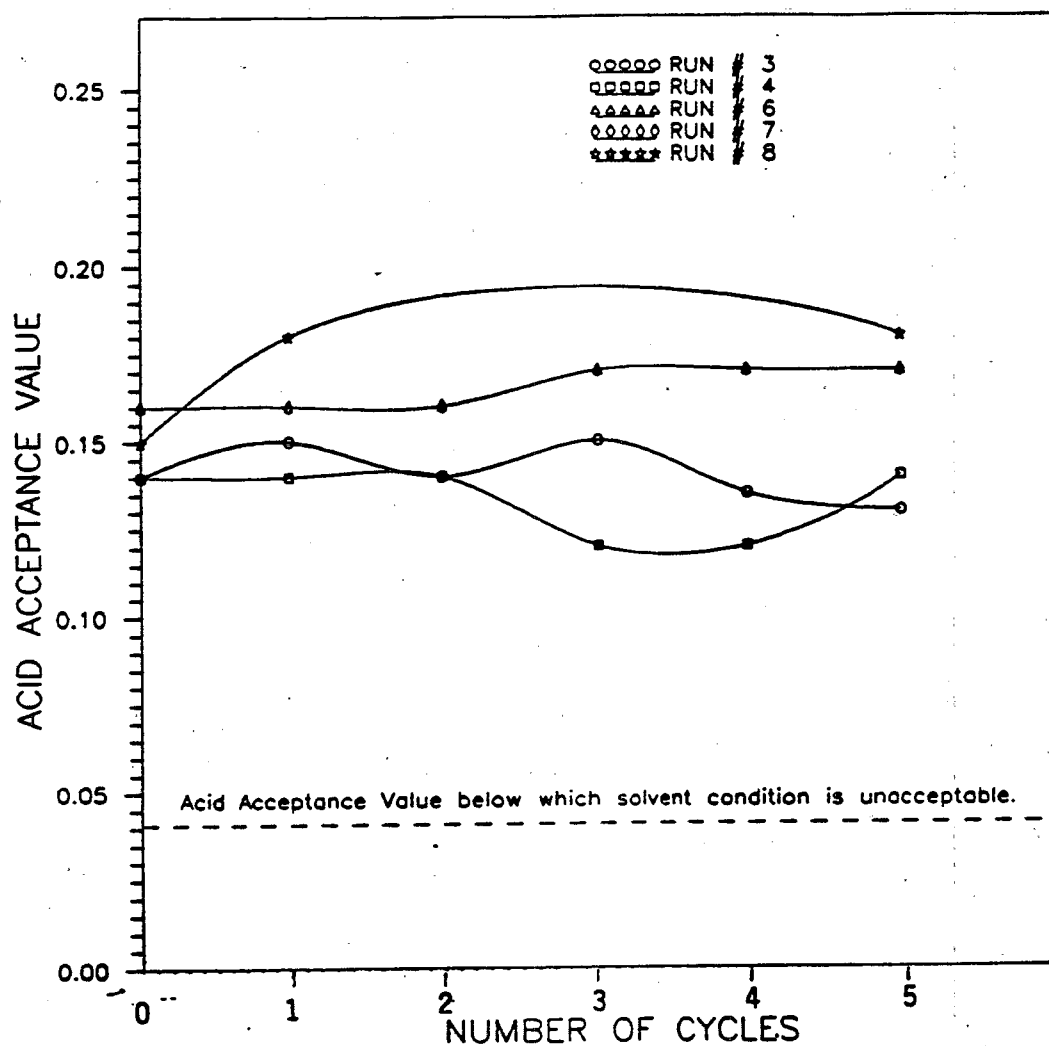


Figure 6. Variation of Acid Acceptance Value With Cycle Times (Set #2)

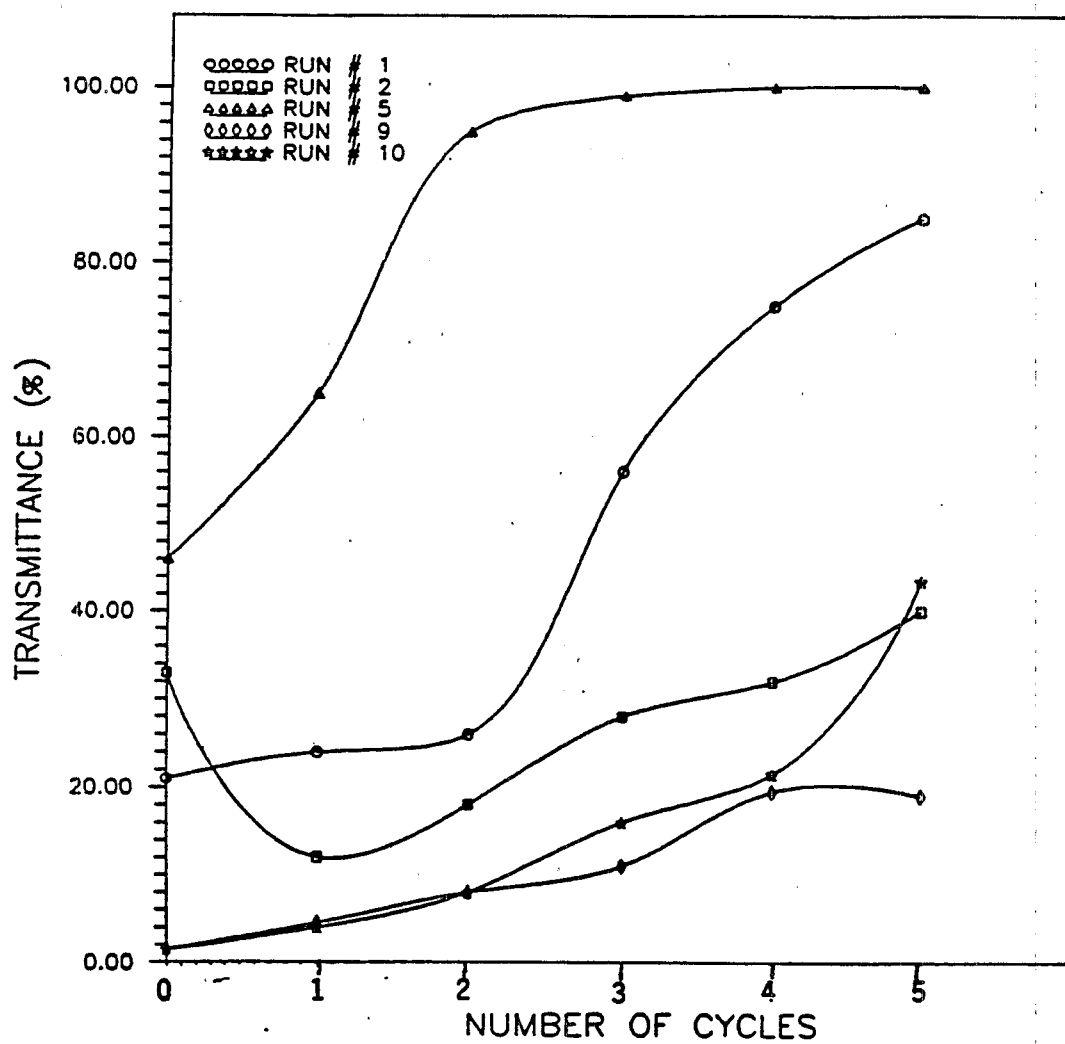


Figure 7. Variation in Transmittance With Cycle Time (Set #1)

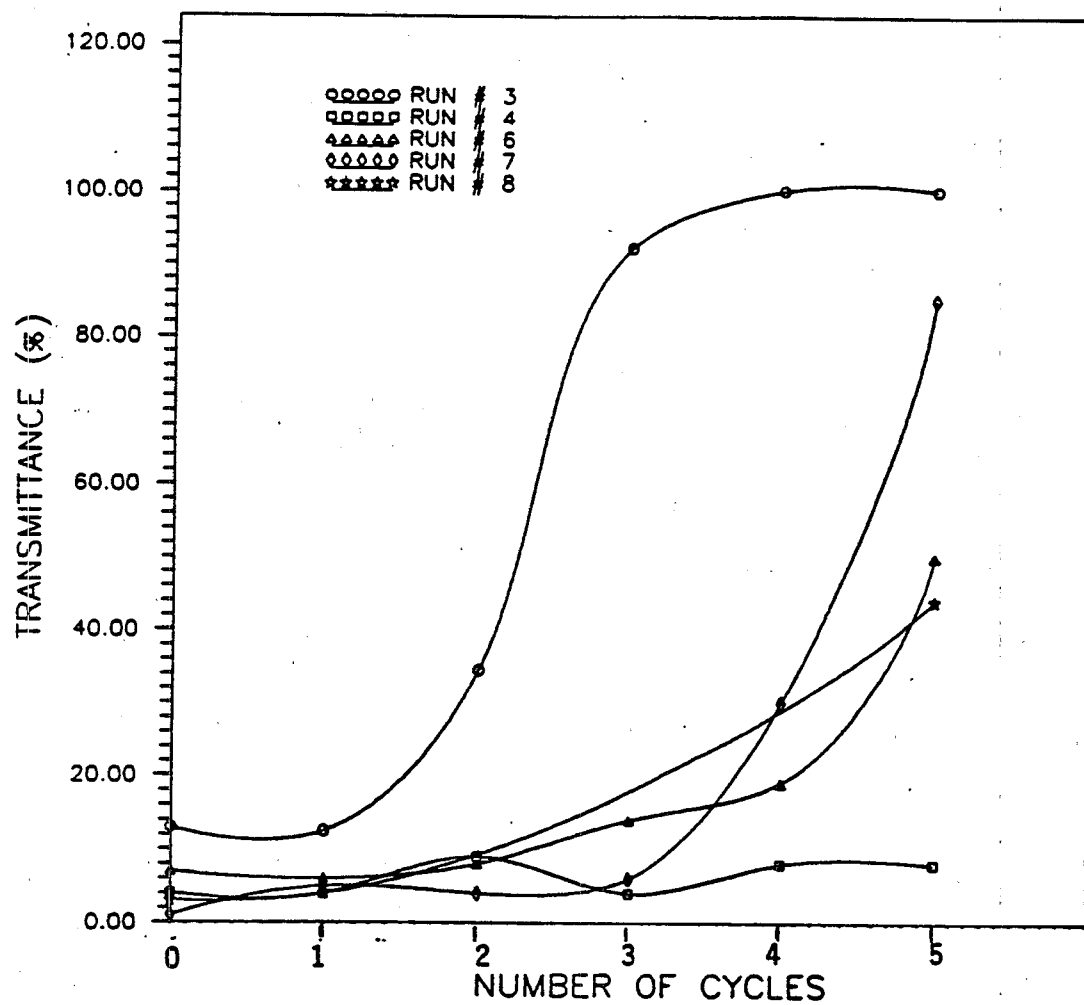


Figure 8. Variation in Transmittance With Cycle Time (Set #2)

B. FILTRATION OF FREON^R 113 (CFC-113)

Used CFC-113 (trichlorotrifluoroethane) was reclaimed by filtration at Warner Robins ALC. The samples taken had high water and solids contents, both of which were reduced by filtration. Water content was reduced by about 90 percent, to very nearly the solubility level of water in CFC-113, which is 0.009 percent.

Solids were almost completely removed, indicating that most of it was particles larger than 0.05 micrometer. Since CFC-113 is primarily used for cleaning delicate precision parts, base personnel thought that the filtered solvent would be usable for preliminary cleaning, if followed by a rinse with virgin solvent.

SECTION V

STABILIZER REPLENISHMENT

If the AAV or GC/MS analysis indicates that solvent is deficient in stabilizer(s), but still acceptable as a cleaner, there are two options that can be used to restabilize it. The first, easiest, and safest is to buy new solvent containing higher than usual levels of stabilizer. This is added to the old bath in quantities sufficient to raise the stabilizer to a safe level. The second option, which requires more skill and training, is to buy stabilizers (often including acid acceptor, metal stabilizer, and antioxidant) and add them as needed. Careful analysis is required to determine the amount to be added, and safety precautions must be strictly observed, as the chemicals used (see Table 2 for some examples) are flammable, toxic, and possibly carcinogenic. For these reasons, only those knowledgeable in and adept at handling chemicals should attempt to make up their own stabilizer packages.

A. DISTILLATION

If, after filtration, the level of dissolved oils and grease is too high, one of the best options for reclaiming solvents of all types is distillation. Properly done, distillation can yield the base solvent in almost virgin condition. Petroleum solvents, such as Stoddard solvent, or PD-680, might require vacuum to lower their boiling points, but are otherwise unaffected by the process. TCA, TCE, and perc all boil at 250°F or below, and may easily be handled in either steam or electrically heated stills at atmospheric pressure. The major disadvantage of distilling halogenated solvents is that the stabilizers could be partially retained in the still bottoms, while the condensate, or overhead, might be understabilized. It is therefore advisable to analyze redistilled solvents to confirm stabilizer levels.

Many stills have been installed, tried a few times, and abandoned because of their presumed difficulty of operation. These could quite likely be reactivated with more thorough operator training and retrofitting with modern automatic control systems. An excellent example of solvent recovery is in operation at Warner Robins ALC, where distillation has been successfully carried out since 1982 (see Reference 7). In 1987, an estimated \$800,000 was saved on CFC-113 alone. (Personnel at the base found that redistilled TCA has retained acid acceptors, but as a precaution, blend the recycled TCA with virgin solvent for safe use. In addition, the base keeps some 1,2-butylene oxide on hand for addition to the reclaimed solvent.)

B. ULTRAFILTRATION

Conventional filtration will normally remove particles of 0.5 micrometer diameter and larger. When particles smaller than 1 micrometer are to be removed, polymeric membranes may be used instead of the solid media of ordinary filters. Some newer membranes are resistant to many solvents, and are capable of retaining molecules larger than 150-200 daltons (molecular weight), which means that Stoddard solvent (ca. 150-160 daltons) or TCA (133.4

daltons) would pass through, but dissolved oils and greases would be retained. The higher-molecular-weight inhibitors such as butoxymethyloxirane (130.2 daltons, see Table 2) would also pass through the membrane. Membrane filters should always be preceded by a conventional filter to minimize fouling. Development of these solvent-resistant membranes, as well as those resistant to acids and bases, is continuing, and membrane separations are expected to replace distillation in many instances.

SECTION VI

MINIMIZING POLLUTION BY MORE EFFICIENT OPERATION

The period between rejuvenation or recycling solvents can be greatly increased by more careful operation of degreasing operations. Szabo and Nutter investigated the technical and economic feasibility of several methods of reducing solvent usage and ambient vapor concentrations near a TCA vapor degreaser at Wright-Patterson Air Force Base (Reference 8). Their recommendations resulted in \$25,200 per year savings in solvent costs, indicating a payback period of 0.6 year. In addition, almost 4000 gallons of TCA were prevented from entering the atmosphere annually.

A similar survey of the various degreasing operations at Tinker ALC was made by a representative of Dow Chemical Co., a major solvent supplier; following those recommendations should save about \$68,000 per year. While the comments made referred to specific units at Tinker, they are applicable to many operations. An edited version of the Dow survey is given below.

A. AUDIT OF DEGREASING BATHS AT TINKER AFB FOR PILOT STUDY

(Note: This section was authored by Jim C. Johnston of Dow Chemical Company, U.S.A., who conducted the audit at Tinker AFB during July 1990. Data given are unchanged; editorial changes were made by Dr. J.D. Wander of Tyndall AFB.)

In this section, each degreasing bath at Tinker AFB is reviewed on its own merit. The second portion of this section estimates solvent savings that could be realized by Tinker if they install steam condensate return pumps, adjust to proper heat balance, use the stop-and-go technique, slow the hoist speed to 2-4 linear feet per minute, and take action on other suggestions made by Dow (Section B following). With these improvements, they could decrease their solvent consumption by not less than 40-60 percent. The calculations are based on a 40-percent solvent reduction (Tables 3 and 4). For the waste solvent of 20-percent contamination (assumed), a projected concentration of 60 percent was calculated for perchloroethylene and 50 percent for 1,1,1-trichloroethane (Tables 5 and 6). For estimation purposes, waste reduction for 5-wt% contamination are also included (Tables 8 and 9), since Tinker AFB appears to be giving to their reclaimer waste that contains less than 5-wt% contamination.

Tinker AFB can achieve a solvent savings of 6,012 gallons plus waste reduction savings of 2,912 gallons; the corresponding annual dollar savings will slightly exceed \$50,000. A solvent reduction of 59.4 percent or greater is achievable (see Table 7).

As Tinker appears to be sending out waste at less than 5 wt%, then even greater savings may be available (see Tables 8 and 9). Table 10 summarizes the solvent and dollar savings that could be realized.

TABLE 3. SOLVENT CONSUMPTION REDUCED BY 40 PERCENT.

Post	Annual Usage (Gals.)	Usage Reduced to (Gals.)	Solvent Usage Reduced to (Gals.)
N-37	1780	1068	712
U-45	1824	1094	730
R-58	654	392	262
W-65	3850	2310	1540
K-35	4180	2508	1672
Ha-49.4	2740	1644	1096
TOTAL	15,028	9,016	6,012

TABLE 4. SOLVENT AND DOLLAR SAVINGS FOR 40-PERCENT SOLVENT REDUCTION.

Post	Solvent	Cost/Gal.	Annual Usage Gals.	Cost	Usage Reduced to (Gals.)	Cost	Sol. Usage Reduced by (Gals.)	Savings
N-45	PCE ^a	\$3.87	1780	\$6889.	1068	\$4133.	712	\$2756.
U-45	PCE ^a	\$3.87	1824	\$7059.	1094	\$4235.	730	\$2824.
R-58	TCA ^b	\$7.74	654	\$5062.	392	\$3037.	262	\$2025.
W-65	TCA ^b	\$7.74	3850	\$29,799.	2310	\$17,879.	1540	\$11,920.
K-35	TCA ^b	\$7.74	4180	\$32,353.	2508	\$19,412.	1672	\$12,941.
Ha-49.4	PCE ^a	\$3.87	2740	\$10,604.	1644	\$6,362.	1096	\$4242.
TOTAL			15,028	\$91,766.	9,016	\$55,058	6012	\$36,708

^a Perchloroethylene

^b 1,1,1-Trichloroethane

TABLE 5. WASTE REDUCTION.

Post	Current Annual Waste* (Gals.)	Waste Concentrated To:	Waste Reduced To: (Gals.)	Waste Reduction %
N-37	480	60%	160	66.7
U-45	420	60%	140	66.7
R-58	468	50%	187	60.0
W-65	990	50%	396	60.0
R-35	1320	50%	528	60.0
Ha-49.4	968	60%	323	66.7
TOTAL	4646		1734	

* Sludge - 20%

TABLE 6. SAVINGS IN WASTE REDUCTION.

Post	Solvent ^a	Cost to Dispose Per Gal.	Current ^b Waste Disposal ^c	Cost	Waste ^d Reduced To:	Cost	Projected Waste Savings	% Waste Reduction
N-37	PCE	\$10.	480	\$4800.	160	\$1600.	\$3200.	66.7%
U-45	PCE	\$10.	420	\$4200.	140	\$1400.	\$2800.	66.7%
R-58	TCA	\$1.	468	\$468	187	\$187.	\$281.	60.0%
W-65	TCA	\$1.	990	\$990.	396	\$396.	\$594.	60.0%
R-35	TCA	\$1.	1320	\$1320.	528	\$528.	\$792.	60.0%
Ha-49.4	PCE	\$10.	968	\$9680.	323	\$3230.	\$6450.	66.8
TOTALS			4646 Gals.	\$21458.	1734 Gals.	\$7341.	\$14,117.	

^a PCE = Perchloroethylene; TCA = 1,1,1-Trichloroethane

^b Sludge - 20%

^c Annually; in gallons

^d In gallons

TABLE 7. TOTAL ANNUAL SOLVENT AND DOLLAR SAVINGS
(20 PERCENT SLUDGE).

TOTAL SOLVENT SAVINGS
(Sludge @ 20%)

	<u>Gals.</u>	<u>Cost \$</u> <u>Savings</u>
Solvent Savings	6,012	\$36,708
Waste Savings	<u>2,912</u>	<u>\$14,117</u>
TOTAL	8,924	\$50,825
Reduce by 40% to:	9,016 Gals/Yr Total Solvent	
Reclaim Reduce Another 30%:	$\frac{-2,912 \text{ Gals/Yr}}{6,104 \text{ Gals/Yr}}$	
Total Reduction of:	$\frac{8,924 \text{ Gals}}{15,028 \text{ Gals}} = 59.4\%$	

TOTAL DOLLAR SAVINGS

Solvent Saving from 40% Reduction:	\$36,708
Projected Waste Savings:	<u>\$14,117</u>
Subtotal	\$50,825
Solvent Reclaimed Value:	<u>\$17,721</u>
Total	\$68,546
Dollar Savings:	$\frac{\$68,546}{\$91,766} = 74.7\%$

TABLE 8. WASTE REDUCTION.

Post	Current* Annual Waste-Gals.	Waste Concentrated To:	Waste Reduced To Gals.	Waste Reduction %
N-37	480	60%	40	91.7
U-45	420	60%	35	91.7
R-58	468	50%	46.8	90.0
W-65	990	50%	99.0	90.0
R-35	1320	50%	132.0	90.0
Ha-49.4	968	60%	81.0	91.7
TOTAL	4646		433.8	

* Sludge @ 5%

TABLE 9. SAVINGS IN WASTE REDUCTION.

Post	Solvent ^a	Cost to Dispose Per Gal.	Current ^b Waste Disposal ^c	Cost	Waste ^d Reduced To:	Cost	Projected Waste Savings	% Waste Reduction
N-37	PCE	\$10.	480	\$4800.	40	\$400.	\$4400.	91.7%
U-45	PCE	\$10.	420	\$4200.	35	\$350.	\$3850.	91.7%
R-58	TCA	\$1.	468	\$468.	47	\$47.	\$421.	90.0%
W-65	TCA	\$1.	990	\$990.	99	\$99.	\$891.	90.0%
R-35	TCA	\$1.	1320	\$1320.	132	\$132.	\$1188.	90.0%
Ha-49.4	PCE	\$10.	968	\$9680	81	\$810.	\$8870.	91.7
TOTALS			4646 Gals.	\$21,458.	434 Gals.	\$1838.	\$19,620.	91.4%

^a PCE = perchloroethylene; TCA = 1,1,1-trichloroethane^b Sludge - 5%^c Annually; in gallons^d In gallons

TABLE 10. TOTAL ANNUAL SOLVENT AND DOLLAR SAVINGS
(5 PERCENT SLUDGE).

TOTAL SOLVENT SAVINGS
(Sludge @ 5%)

	Cost/ Gals. (\$)	Savings (\$)
Solvent Savings	6,012	36,708
Waste Savings	<u>4,212</u>	<u>19,620</u>
TOTAL	10,224	56,328

Solvent Consumption Reduced by 40%: 9,016 Gals/Yr

Reclaim Solvent from Waste: -4,212 Gals/Yr

Gals. Needed for the Coming Year: 4,804 Gals/Yr

Total Solvent Reduction = $\frac{10,224 \text{ Gals.}}{15,028 \text{ Gals.}}$ = 68%

TOTAL DOLLAR SAVINGS

Solvent Savings from 40% Reduction: \$36,708

Projected Waste Savings: +19,620

SUBTOTAL: \$56,328

Solvent Reclaim Value: +25,983
\$82,311

Dollar Savings = $\frac{\$82,311}{\$91,766}$ = 89.7%

TABLE 11. HALOGENATED SOLVENTS USAGE INFORMATION.

Post	Unit Size	Solvent	Batch Changes	ATG Add/Wk	Batch Vol/Gal	Batch Temp.(°F)	Reservoir Vol.	Annual Usage*	Annual Waste	Emiss. Loss
N-37	5.3x3.4x5.4	Perchloroethylene	6	25	80	250-260	35	1780	480	73.0%
U-45	5.0x2.5x6.5	Perchloroethylene	17	27	70	250-260	50	1824	420	77.0%
R-51	4.0x4.5x1.7	Trichlorotrifluoroethane	26	2	30	110-115	--	884	468	47.1%
N-58	4.5x3.0x5.6	1,1,1-Trichloroethane	10	2	55	165-175	50	654	275	58.0%
W-65	8.0x4.0x8.0	1,1,1-Trichloroethane	6	55	165	165-175	110	3850	990	74.3%
R-35	10.0x3.0 x10.	1,1,1-Trichloroethane	8	55	165	165-175	110	4180	1320	68.4%
Ha-49.4	8.0x4.0x8.5	Perchloroethylene	4	40	165	250-260	110	2740	968	64.7%

B. SURVEY OF TINKER AFB DEGREASERS

1. Cooper Open-Top Degreaser N-37

Solvent: Perchloroethylene

Suggestions:

a. Use a steam condensate return pump to return the steam condensate instead of using the steam pressure to return the condensate to the boiler. The use of excessive steam pressure results in boiling the solvent faster, but not hotter. This results in higher solvent emission loss in the range of 7-15 percent.

b. If this machine is to be used in this area continuously, door strips or welder strips should be hung in the doorway or around the degreaser to reduce the drafts.

c. The cold trap was iced up. It would help to raise the temperature of the refrigeration unit to 0° or 20°F. This would decrease the volume of water condensing into the degreaser.

d. The water separator was nonfunctional. It contained approximately 3 inches of water which needed to be expelled immediately. This can be accomplished by restricting the solvent flow back into the degreaser by installing a needle valve in the exit line. By use of the needle valve, the solvent flow can be reduced to raise the solvent level to a quarter or half-inch below the center line of the water drain outlet. The water drain spigot would be left open all the time so the water could be drained constantly into a 5-gallon open container. By law (the Clean Water Act), you cannot allow any of the water to fall on the ground or contaminate the ground water. The other way the water from the top of the water separator can be removed is to siphon the water layer off daily.

e. Lower the hoist speed to 2 linear feet per minute. Holding your hoist speed in the range of 2 to 8 linear feet per minute can decrease solvent losses by 7-27 percent. The lower the speed, the lower the solvent emissions.

f. Make sure your cooling water is coming in at the bottom of your condensing coils. This prevents condensation of water. To get the desired effect, the cooling water must exit from the top of the coil.

g. Keep degreaser covered when idling or not in operation. *This unit was covered properly while in the idling mode.*

h. Have operators use the stop-and-go technique. This technique can decrease solvent consumption by 12-27 percent.

i. Use a smaller work basket, so it takes up only 50 percent of the freeboard area. This will decrease the plunger effect and greatly lessen solvent loss due to plunging, chimney, and drag-out effects. Using a properly sized workload basket can contribute to a savings of solvent from 10-20 percent.

2. Open-Top Degreaser U-45

Solvent: Perchloroethylene

This machine was expelling abnormally high vapor fumes which appeared to be the result of situations described below. These suggestions were made:

a. Heat balance: Excessive heat input. Degreaser should operate on 45-50 psi. See item a on degreaser N-37 regarding installing steam condensate return pump.

b.. It appeared that there was also a water flow problem through the condensing coils and water jacket. Possibility of Penn Control valve not properly adjusted or malfunctioning. The exit water was so hot that you could not hold your hand on the pipe. Entrance water should be between 50°-60°F. and exit water between 90°-100°F. Probably good reason for having a water chiller and a closed-loop cooling system.

c. The condensation troughs were backed up, which could be a result of over-heating, partial plugging of the drain lines from the degreaser or water separator, a valve partially closed in the solvent return line, or a combination of these possibilities.

d. It would also be a good idea when you need to replace the condensing coils to use at least 1/2 - 5/8 inch diameter coils for better vapor control.

e. Refer to item e, degreaser N-37 (Hoist speed).

f. Refer to item h, degreaser N-37 (Stop-and-Go technique).

3. Delta Open-Top Degreaser HA-49.4

Solvent: Perchloroethylene

Suggestions:

a. Heat balance overheating: see item a, degreaser N-37. Steam pressure should be about 45-50 psi.

b. Coil trap was frosted: refer to item c, degreaser N-37.

c. Water separator nonfunctional: check to see if water drain spigot is open and if solvent level is within 1/4 to 1/2 inch of the center line of the spigot. Refer to item d, degreaser N-37.

d. Check Hoist speed. Refer to item e, degreaser N-37.

e. Condensing water: refer to item f, degreaser N-37.

f. See item i, degreaser N-37 (basket size).

g. Stop-and-Go technique: refer to item h, degreaser N-37.

h. Keep degreaser covered: refer to item g, degreaser N-37.

4. Ramaco Open-Top W-65

Solvent: 1,1,1-Trichloroethane.

Suggestions:

a. Incorporate a water chiller to give year-around water at 50°F. This will give you a closed loop. The closed loop will alleviate your internal water recycle problem.

b. Install a steam condensate return pump: refer to item a, degreaser N-37.

c. Water separator nonfunctional: refer to item d, degreaser N-37.

d. Hoist speed: refer to item e, degreaser N-37.

e. Change water inlet to bottom of condensing coil. Refer to item f, degreaser N-37.

f. Keep degreaser covered when idling or not in operation. The unit was properly covered at time of inspection.

g. Stop-and-Go technique: refer to item h, degreaser N-37.

5. Phillips Open-Top R-35

Solvent: 1,1,1-Trichloroethane

Suggestions:

a. Unit was being overheated. Use steam condensate return pump (refer to item a, degreaser N-37) so that the steam pressure can be reduced to 3-4 psi or lower steam pressure.

b. Water separator was not functioning: refer to item d, degreaser N-37.

c. There is a problem with solvent backup in the condensation trough. Refer to item c, degreaser U-45.

d. Water was noted floating in the condensation trough. This could be due to a water leak in the condensing coil or the result of water picked up from the atmosphere separating in the trough because the trough is not emptying properly.

e. Again, good candidate for a water chiller to prevent having to use excess steam pressure. Refer to item b, degreaser U-45.

f. Check to see that inlet cooling water is coming in at bottom of condensing coil. Refer to item f, degreaser N-37.

g. The plastic cover was in place, but had numerous holes in the top.

h. Check the hoist speed. It should not be greater than 2-3 linear feet per minute. Refer to item e, degreaser N-37.

i. Have operators use Stop-and-Go technique. Refer to item h, degreaser N-37.

6. Ramaco Open Top Degreaser N-58

Solvent: 1,1,1-Trichloroethane

This machine was in good condition but needed the following adjustments to reduce solvent emission:

a. Install steam condensate return system. Refer to item a, degreaser N-37. This unit can normally operate on 1 to 1-1/2 psi.

b. Use a water chiller: refer to item a, degreaser W-65.

c. Hoist speed: refer to item e, degreaser N-37.

d. Water separator was nonfunctional: refer to item d, degreaser N-37.

This machine had its top in place at time of inspection.

SECTION VII

CONCLUSIONS AND RECOMMENDATIONS

Recovery by purification and distillation and in-situ filtration are practical options to extend the effective working lifetime of metal-cleaning and degreasing solvents when used in conjunction with a program to monitor levels of protective additives and maintain additive protection above critical thresholds. Refinement of operating technique and aggressive maintenance policies can decrease the rate of loss of solvent from degreasing baths.

Refinement of in-situ purification technology and implementation as a field test is recommended. As part of a quality control program, degreaser operators should be retrained at regular intervals, with unscheduled evaluations of operating technique on the line and of maintenance status of the degreasing baths performed.

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